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Infrared sensing in the two wavelength ranges (3-5 um and 8-12 um) has important military applications. Most commonly used detecting materials and devices are HgCdTe-based detectors and quantum-well infrared photodetectors (QWIP). Antimonide-based III-V materials also have potentials. To further realize a multi-wavelength imaging device, however, requires the integration of infrared photodetector array consisting of materials having defferent bandgap energies (usually having different lattice dimensions too). A major challenge arises - to obtain high-quality low-defect heteropitaxial layers.

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Final Report

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"Surface Engineering for Compliant Epitaxy"

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Table of Contents

Research Objectives and Technical Approaches

Infrared sensing in the two wavelength ranges (3-5 μm and 8-12 μm) has important military applications. Most commonly used detecting materials and devices are HgCdTe-based detectors and quantum-well infrared photodetectors (QWIP). Antimonide-based III-V materials also have potentials. To further realize a multi-wavelength imaging device, however, requires the integration of infrared photodetector array consisting of materials having different bandgap energies (usually having different lattice dimensions too). A major challenge arises – to obtain high-quality low-defect heteroepitaxial layers. In general, large mismatch tends to induce a high density of misfit and threading dislocations during heteroepitaxy leading to device degradation, which is the limiting factor in the monolithic integration. There are two possible approaches that can be employed to realize these highly strained but low-defect materials, i.e. compliant epitaxy and metamorphic heteroepitaxy, and extensive research is needed.

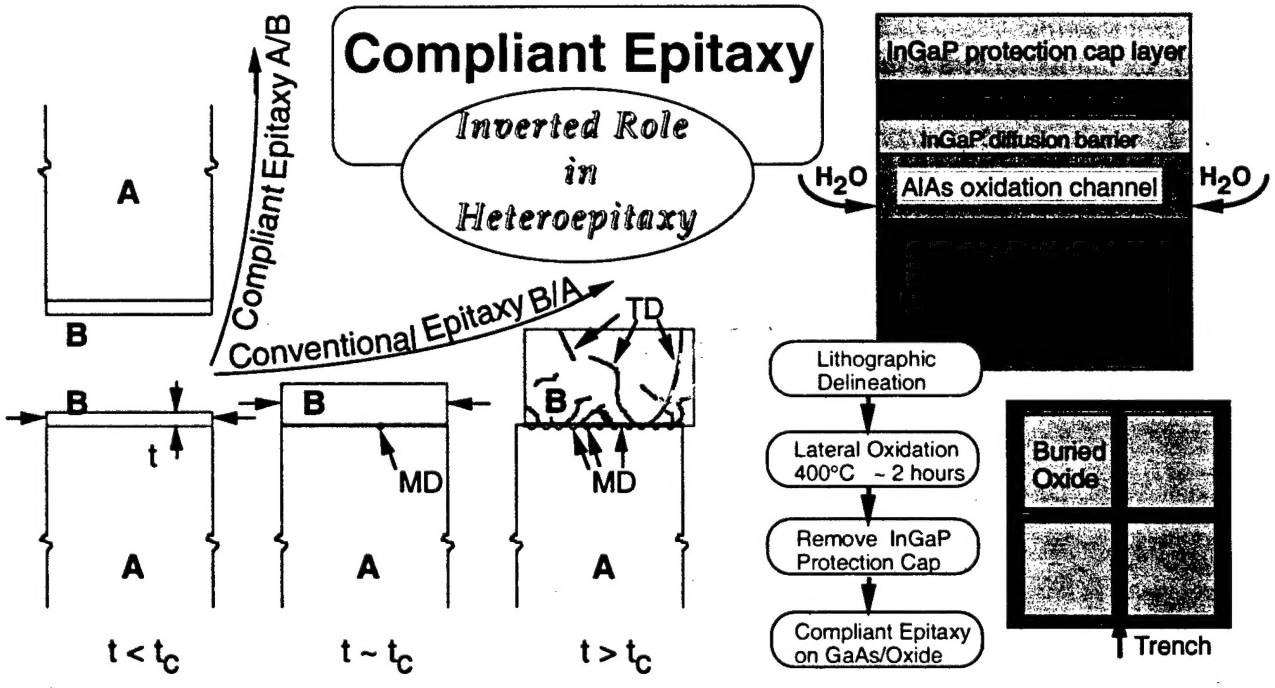
Our research objectives are (1) to develop a selective area compliant template and (2) to develop strain-relieved template on which InP-based lattice mismatched epitaxy can be realized with low defect density. We have conducted engineering processing related to lateral oxidation in order to realize low-defect high-quality strained layer. A strained system of InGaAs on GaAs is our test vehicle to verify the underlying principle for the proof-of-concept. The two technical approaches, (1) surface engineering for strain-absorbing compliant epitaxy and (2) oxidation-induced defect reduction in strain-relieving metamorphic heteroepitaxy, and our accomplishments are illustrated as follows.

II. Accomplishment

(1) *Surface engineering for strain-absorbing compliant epitaxy*

As illustrated in the figure, compliant epitaxy plays an inverted role in terms of defect generation as compared to conventional strained layer heteroepitaxy. In conventional heteroepitaxy, where the substrate is practically infinitely thick, the misfit strain can no longer be accommodated elastically when the epi-layer exceeds the so-called critical thickness, t_c. As a result, misfit and threading dislocations form in the epi-layer. However, for films thinner than t_c, there are no dislocations, and all strain is elastically stored in the thinner epi-layer. There is no difference in viewing the heteroepitaxy as thin epilayer grown on thick substrate or oppositely thick substrate grown on thin epilayer template. The net result is that two films having a large thickness difference are epitaxially attached.

A simple paradigm shift can easily enable one to visualize compliant epitaxy as an inverted heteroepitaxy, i.e. a switched role between substrate and epilayer. In compliant epitaxy, it is the thin template, which deforms compliantly to absorb all strain energy. A significant reduction of dislocations can be realized by performing heteroepitaxy on compliant substrates as most misfit strain is absorbed in the compliant substrate instead in the epitaxial layer.



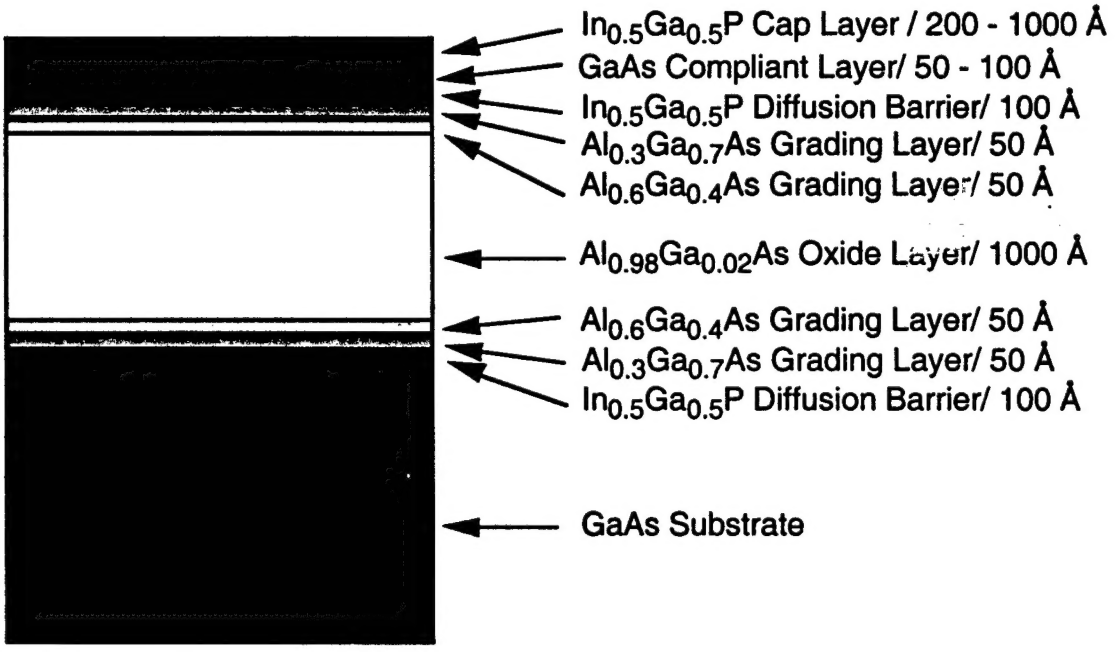
Fundamentally speaking, the key to ensuring compliance is to realize a thin substrate (or template), the thinner the better. Our technical approach to forming a thin compliant GaAs layer for subsequent mismatch epitaxy is to employ lateral wet oxidation of an underlying Al-bearing compound while leaving intact a thin GaAs overlayer. On the thin GaAs template, we then deposit highly strained InxGa1-xAs layer. Technical challenges are: how to realize

thin and sturdy template, how to obtain pinhole free template, and what is the maximum dimension of template for compliant epitaxy.

(A) Design of Compliant Template

We have developed a thin GaAs template (~100Å) on buried oxide. The buried oxide is formed by wet oxidation of the underlying AlGaAs layer on which the thin GaAs template is grown. Wet oxidation of AlGaAs has been found sensitive to many factors including layer thickness, aluminum content, oxidation temperature, moisture content. Once oxidation is completed, residual stress within the oxide sometimes leads to layer delamination such that the layer on top of the oxide tends to peel off. The peel off adds complexity to subsequent processing. In addition to residual stress, back stream oxidation up towards surface, although slower in rate, eventually fatally renders the thin GaAs compliant layer to become amorphous during the lateral oxidation process. No high quality single crystal has ever been deposited on an amorphous template. So stress management and implementation of oxidation barrier is critical to the design of compliant template on buried oxide by wet oxidation. We have completed an excellent design of compliant template and demonstrated the growth of lattice matched epitaxy on compliant template over 50-μm stripes.

To develop a robust template that can stand stress-induced peeling and back-stream oxidation, we have realized a structure incorporating (i) Graded structure from high Al content oxide channel to neighboring GaAs, (ii) InGaP as an oxidation barrier, and (iii) Digitized $Al_{0.98}Ga_{0.02}As$ oxide channel. A schematic diagram of this structure is shown as follows.



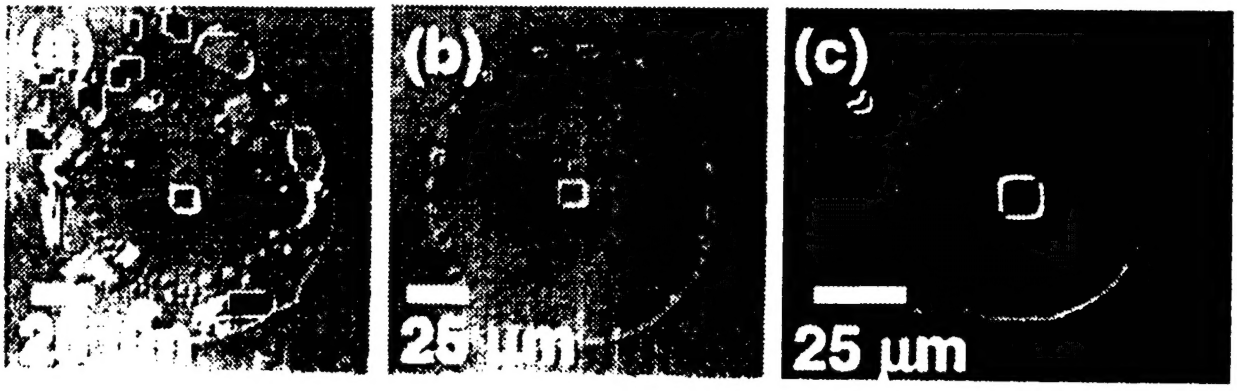
(i) Graded AlxGa1-xAs structure and Incorporation of InGaP as an oxidation barrier
Oxidation rate of AlGaAs depends monotonically on the Al content. Pure AlAs has the highest oxidation rate. A sharp transition from pure AlAs to GaAs, however, presents some challenges to stress management when the AlAs layer is oxidized. The sharp transition gives rise to weakest bonding between the oxide and crystalline GaAs, often leading to

delamination. From mechanics point of view, it delaminates in a brittle manner. To minimize that, a mechanism to increase the ductility is desired. We have used a graded structure from high Al content to GaAs since oxidation drastically slows down with a lower Al content. We have found that a step-graded structure from AlAs to $Al_{0.6}Ga_{0.4}As$ to $Al_{0.3}Ga_{0.7}As$ to GaAs is effective to reduce delamination.

Although GaAs is rather inert to wet oxidation as compared to AlAs, it does not stop the oxidation. In comparison, InGaP is even more inert to oxidation. We have measured the oxidation rate of InGaP and found that it is only 1/3 to that of GaAs. Since InGaP can be grown lattice matched to GaAs with proper In and Ga contents, it is rather advantageous to incorporate InGaP as an oxidation barrier. Results show that InGaP is very effective to stop wet oxidation either from top or back stream from bottom when lateral oxidation is performed at the temperature range from 400 to 450 C usually taken in our experiments. It is found that for wet oxidation less than an hour at 425 C, InGaP of 50Å is thick enough to protect GaAs from oxidation. The thickness may need to increase accordingly if higher temperature or longer period of oxidation is performed.

(ii) Digitized $Al_{0.98}Ga_{0.02}As$ oxide channel

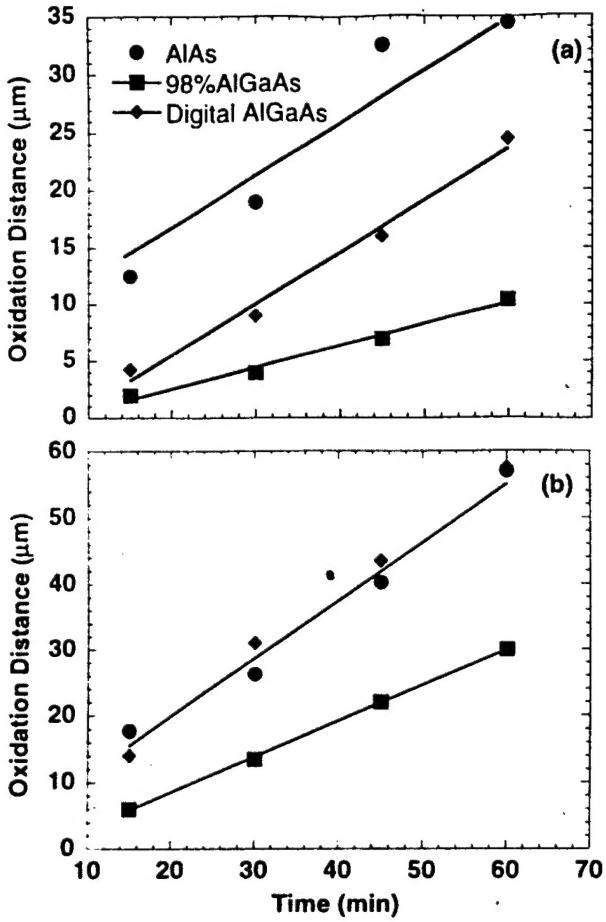
It is found that the oxidation rate of AlGaAs is very sensitive to the Al content especially when it approaches pure AlAs. For example, a mere 2 % decrease in Al content may reduce the oxidation rate in half. However, it is also found delamination is also more severe for GaAs on oxide resulted from pure AlAs. In contrast, a much-improved morphology of GaAs can be realized if a small amount of GaAs is added to the otherwise pure AlAs oxidation layer. A comparison is shown in the following figure.



Nomarski photographs of the laterally oxidized a) pure AlAs, b) digital $Al_{0.98}Ga_{0.02}As$ and c) $Al_{0.98}Ga_{0.02}As$ samples after being heated at 450 °C for 3.5 minutes. The GaAs cap in the pure AlAs sample has delaminated from the underlying oxide.

Compromising among the oxidation rate and induced stress, we have chosen $Al_{0.98}Ga_{0.02}As$ to be the oxidation channel. A tight control of the Al content of the oxidation channel to be exactly about 98% is unfortunately not easy although it is critical to the control of oxidation rate. One of the major reasons is the accuracy of temperature control of the Knudsen cell in an MBE system. A few degree change from run to run and day to day operation is common, which may result in one or two percent change in the Al content of an AlGaAs layer easily.

To overcome the uncertainty, we have employed a short period superlattice structure consisting of one monolayer of GaAs and 50 monolayers of AlAs. Since each constituent of GaAs and AlAs is binary, stoichiometry is not a concern anymore. In addition, the growth rate of GaAs and AlAs can be accurately monitored with the RHEED intensity oscillation technique. The short-period superlattice structure then gives rise to an average composition of $Al_{0.98}Ga_{0.02}As$. We have also found that the lateral oxidation rate remains the same in the $Al_{0.98}Ga_{0.02}As$ channel as pure AlAs if superlattice structure is used instead.

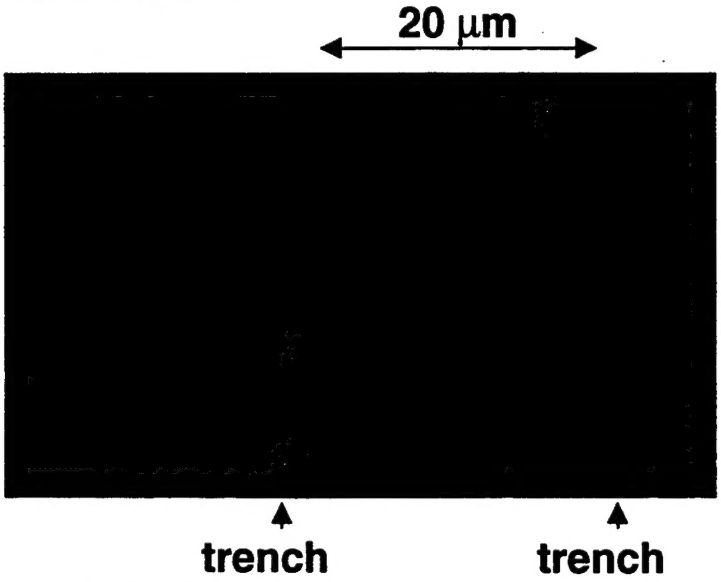


Lateral oxidation distance versus time for AlAs, $Al_{0.98}Ga_{0.02}As$ and AlAs/GaAs digital alloy sandwiched between GaAs layers at a) 425 °C and b) 450 °C. Notice that the oxidation rates of the pure AlAs and the digital alloy are the same and are twice the rate of the $Al_{0.98}Ga_{0.02}As$ alloy.

(B) Epitaxy on compliant template

B-1: Lattice-match epitaxy

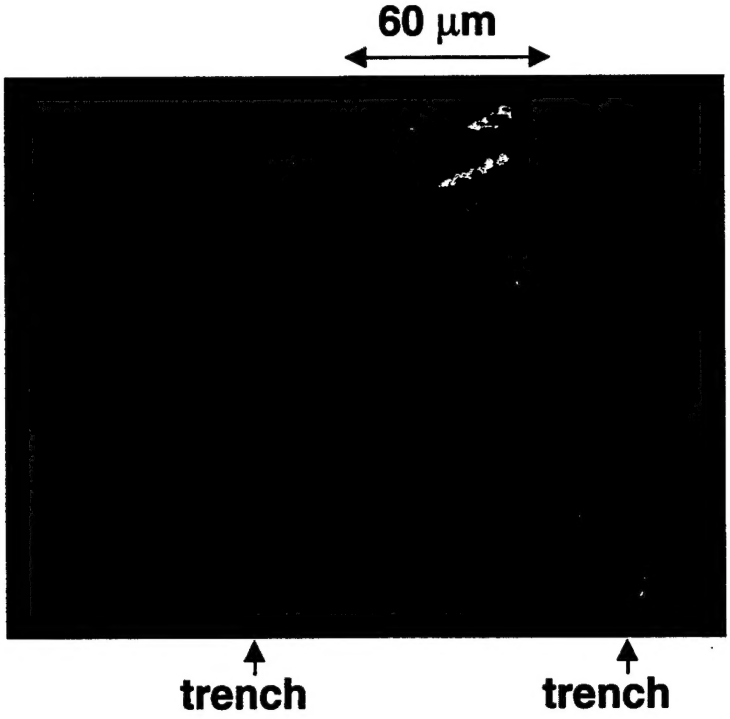
We have grown GaAs/ $Al_{0.3}Ga_{0.7}As$ superlattice on the compliant template to evaluate the quality of the complaint template. The compliant substrate is obtained by opening 5μm trenches on a 20-μm spacing with standard lithographic technique. It is then followed by wet oxidation at 400 or 425 C of the ordinary $Al_{0.98}Ga_{0.02}As$ alloy channel. After preferentially etching off the InGaP cap layer with HCl, a thin GaAs seeding layer bonded to the underlying InGaP oxidation barrier which in turn is bonded to the oxide channel, is obtained. The substrate is then loaded into the MBE chamber for the regrowth of GaAs/AlGaAs superlattice onto the thin complaint GaAs seeding layer after the surface oxide has first been desorbed at 600 C in vacuum.



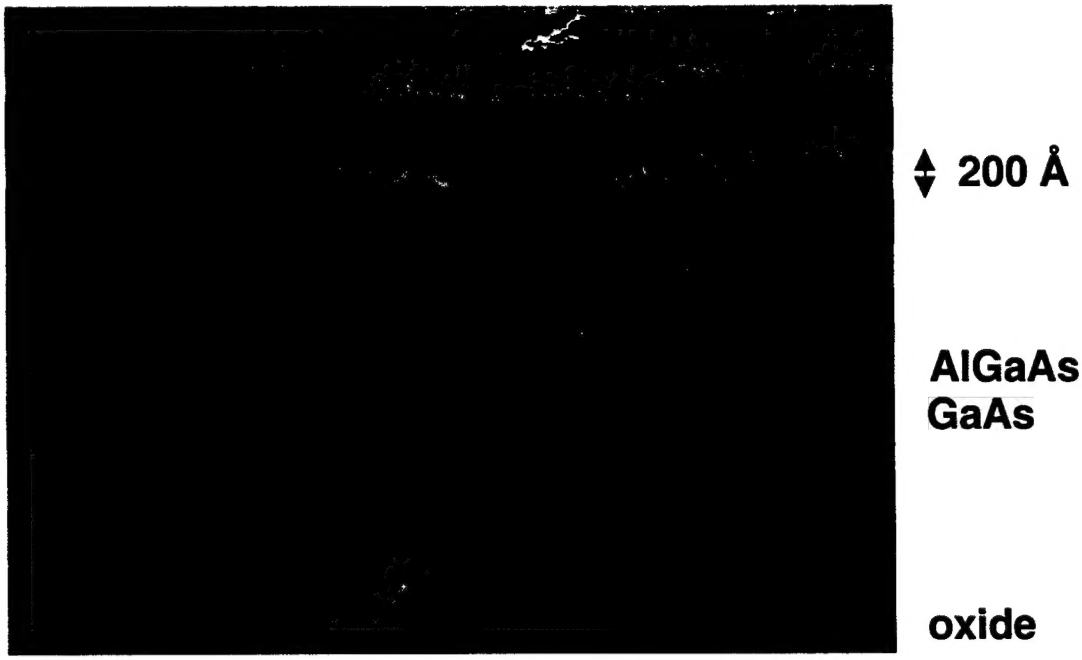
Optical micrograph (above) shows that the morphology of the regrown superlattice is not uniform. It is smooth in all trenches, which are of 5 μm in width. Between two trenches is a 20-μm stripe. On the 20-μm stripe, the morphology is smooth in the central 10-μm region while it looks rough on both rims near the open trenches as shown below. TEM shows that poor morphology corresponds to defective crystallinity, although single crystalline. The poor growth near the edges is attributed to stress nonuniformity and existence of pinholes near the edges of the compliant template, both closely related to the differential in wet oxidation laterally versus vertically upwards. It is very likely that vertical oxidation, though slower because of the graded structure and InGaP barrier layer, continues as long as lateral oxidation proceeds. As a result, a more extensive back stream upward oxidation concentrates on regions near the edges of the stripes than laterally deep inside the stripe. Consequently, there might be pinholes in the thin GaAs seeding layers near the edges if over oxidation occurs. It is then likely to initiate poor crystalline structure near the edges of the stripes. To circumvent the problem, a mechanism to enhance the lateral oxidation while suppressing the vertical is needed, i.e. to biasedly increase the differential between lateral and vertical oxidation.

Previous results on oxidation of digitized $Al_{0.98}Ga_{0.02}As$ indicate that the digitized channel can oxidize laterally just as fast as pure AlAs providing the much needed lateral/vertical rate differential. We have then grown a high quality GaAs/AlGaAs superlattice on the new and

improved compliant template whose oxidation channel is made of digitized $Al_{0.98}Ga_{0.02}As$. Optical micrograph shows that rough morphology near the edges of stripes no longer exists. In fact, growth on 60/40 μm patterned stripes yields smooth morphology over the 60-μm stripes, as shown below, a size adequate for device fabrication.



TEM results (below) indicate that sharp AlGaAs/GaAs superlattice is obtained. The crystalline quality has been further confirmed with photoluminescence measurement. Very high luminescence obtained from the superlattice manifests not only the excellent crystallinity but also the high device quality.



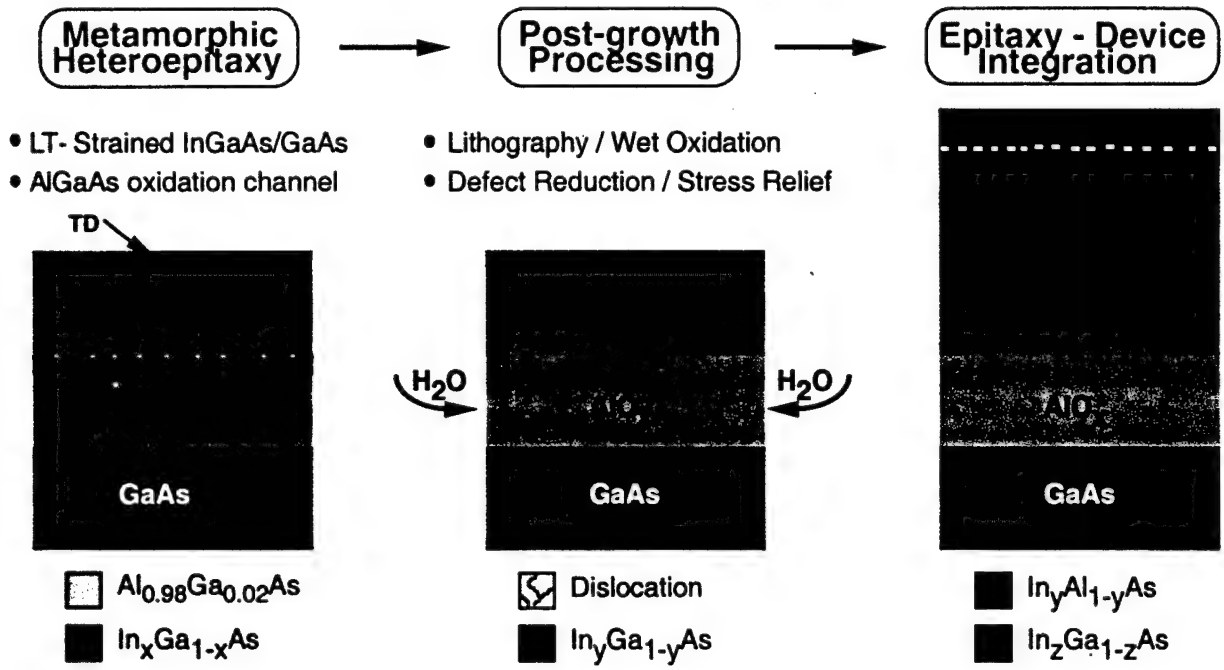
B-2: Lattice-mismatch epitaxy – Assessment of compliant heteroepitaxy

Growth of InxGa1-xAs on our best-prepared compliant template did not yield the much-anticipated defect-free strained layer epitaxy. The dislocation density appears to be about the same order as those encountered in the conventional strained layer heteroepitaxy. There are two possible causes for the noncompliance. (i) One of them is that the 100Å template is still too thick so it is still too rigid to deform elastically before the misfit dislocations form in the InGaAs layer to relieve the strain. A simple remedy can be found by reducing the GaAs template further to about 50 or even 25 Å provided the crystalline quality of the template can be preserved. As the thickness of the template reduces it becomes more challenging to prevent the underlying back-stream vertical oxidation from reaching under and rendering the crystalline quality of the GaAs template. (ii) The second possible cause is much more fundamental than technological. The lack of compliance may be due to the strong bonding between the thin GaAs template and the underlying oxide although the oxide is somewhat porous and not as strong and dense as a single crystal aluminum oxide (corundum). Among three possible bonding forces, covalent, hydrogen and Van der Waals bonding, Van der Waals is the weakest, which is about 1/10 of the covalent chemical bond. Nevertheless, it is still very strong and maybe simply too strong to let the overlaying GaAs template deform freely.

Should the strong bonding between the template and oxide be the reason for compliance lacking, compliant heteroepitaxy can only be preformed on a completely freestanding thin template. It is only possible to conduct such experiments in a gravitation free environment such as in the outer space. For practical reasons, we have decided to concentrate our effort on achieving high quality low-defect heteroepitaxy by the strain relieving defect-reduction approach.

(2) *Oxidation-induced defect reduction in strain-relieving metamorphic heteroepitaxy*

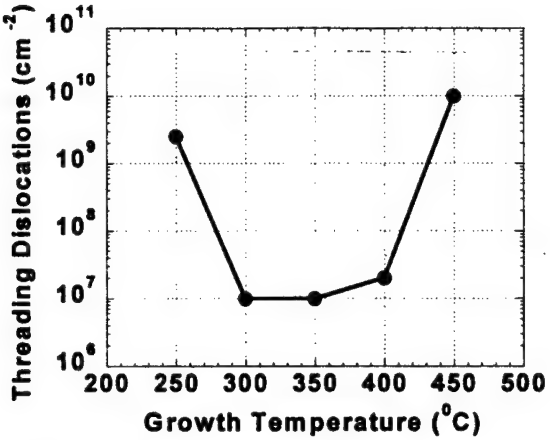
Metamorphic epitaxy is a growth technique that employs a series of grading steps, each with a higher mismatch. It can be a series of steps in composition difference or in the extreme case, a linear change in composition. In general, it is performed at lower temperatures. As a result, misfit dislocations form in bands as the surf
certain critical value, and a relatively small amount of threading dislocations appear near the surface. It has been found that a relatively defect-free InxGa1-xAs (x>50%) film can be realized on GaAs substrate with a defect density in the order of 10^6~10^7/cm^2. To further reduce the dislocation density we have laterally oxidized an underlying Al-bearing compound, as shown in the figure, because wet oxidation has been known to induce strain



relaxation.

(1) Low temperature metamorphic epitaxy for In0.25Ga0.75As with low density of threading dislocations

Instead of employing a series of grading steps of linear grading, we have performed the strained layer heteroepitaxy of In0.25Ga0.75As in a single step as in a conventional heteroepitaxy. In general, a high density of defects including misfit and threading dislocations is expected when the epitaxial layer exceeds the critical thickness. However, the density of defects depends closely on the growth conditions. Of greatest interest to device applications are films with complete strain relaxation and least threading dislocations. We have grown In0.25Ga0.75As film on GaAs with a low density of threading dislocations by employing low growth temperature and low arsenic overpressure. As shown in the following figures, the defect density can increase by three orders of magnitude if the growth conditions are not optimized. As the film thickness increases, it is also found that the strain relaxation approaches 100% and the threading dislocations increases from zero to a saturation value as low as 10^7/cm^2.



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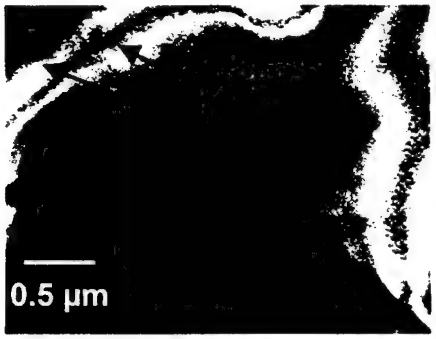
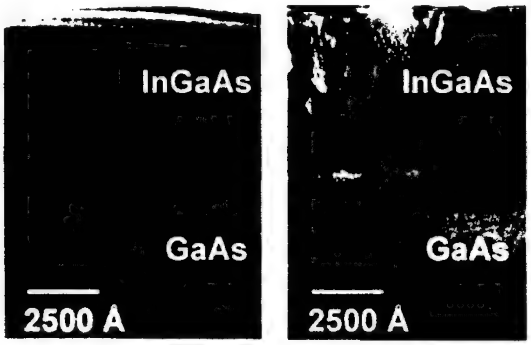


a) Threading dislocation density versus growth temperature for 5000 Å $In_{0.25}Ga_{0.75}As$ epilayers grown on GaAs. Note a minimum in the threading dislocation densities for growth temperatures in the range of 300 – 350 °C. b) Plan-view transmission electron microscopy micrograph of a 5000 Å $In_{0.25}Ga_{0.75}As$ epilayer grown at 350 °C. A threading dislocation density of ~10^{7} cm^{-2} was measured for this sample.

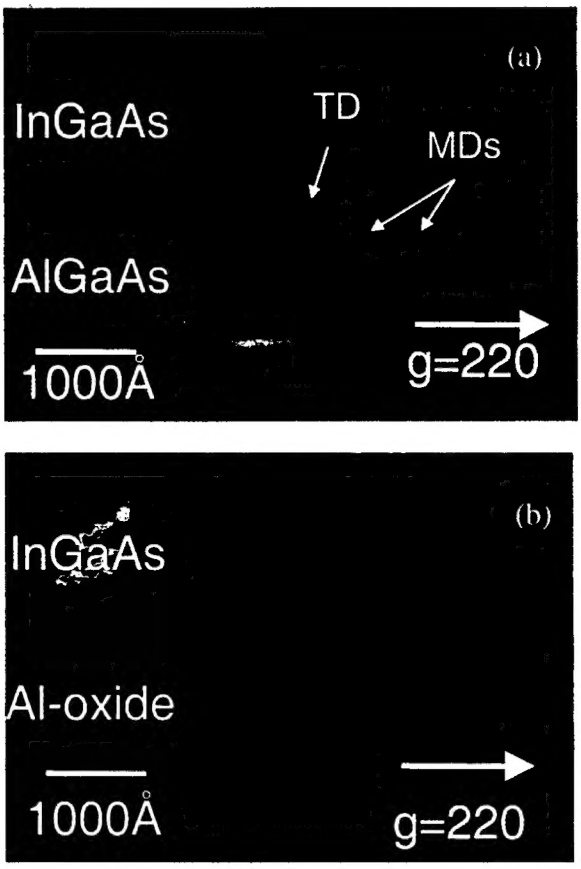


a) b)

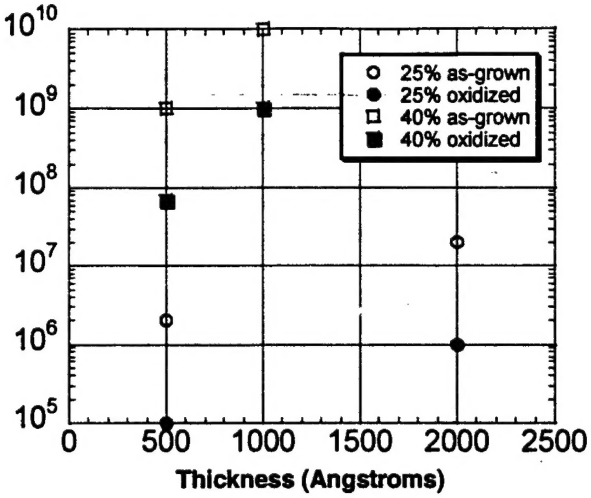
Cross-sectional transmission electron microscopy micrographs of a 5000 Å $In_{0.25}Ga_{0.75}As$ epilayer grown at a) 300 °C and b) 450 °C. Note the dramatic difference in the number of threading dislocation in the layer grown at the lower temperature.

(2) Oxidation-induced defect reduction

We have performed lateral oxidation of an underlying AlGaAs channel and found that the threading dislocations in the overlaying In0.25Ga0.75As strained layer reduces by one order of magnitude as shown in the following figures. It is understandable that the interfacial misfit dislocations disappear as a result of material loss in the Al0.98Ga0.02As digitized channel during lateral oxidation. It is less clear as to how the threading dislocations are also removed during the oxidation process. It is believed that the material loss and hence a volume reduction in the oxidation channel leads to a large stress in the oxidation front. As the lateral oxidation proceeds toward the interior of the stripes, the misfit dislocations are driven either toward the edge and disappear. Alternatively, the movement of threading dislocations enhances the interaction among them and some of them annihilate each other when they have the same Burgers vectors leading to the density reduction. Most importantly, this enabling technique opens a door to realizing low-defect strain-relaxed epitaxial growth.



TEM micrographs of (a) as-grown 2000 Å In0.25Ga0.75As strained layer with underlying AlGaAs oxidation channel and (b) lateral oxidation of the AlGaAs channel. The micrographs indicate that all misfit dislocations (MDs) are removed by the oxidation process and the amount of threading dislocations (TDs) decreases.



Threading dislocation density in the InxGa1-xAs layer (x=0.25 and 0.4) of the as-grown and oxidized samples. The density of threading dislocations in the InxGa1-xAs layer decreases by approximately one order of magnitude after the underlying AlGaAs layer is oxidized. For 500 Å In0.25Ga0.75As, the density of threading dislocations is estimated to be below $10^5/cm^2$.

(3) Protection of In0.25Ga0.75As against oxidation

One of the critical requirements to obtaining high quality crystal growth by molecular beam epitaxy technique is preserving and maintaining a clean surface of the substrate. Usually it is accomplished by desorbing the weak surface oxide in an ultrahigh vacuum environment. However, an unprotected surface of the InGaAs layer develops a tough oxide during the harsh lateral oxidation process of the underlying AlGaAs channel. It is difficult to desorb the tough surface oxide in MBE and subsequent growth results in poor crystal films. A common practice to circumvent the problem is to apply a protection cap layer on top of the InGaAs layer. This encapsulating layer such as PECVD SiO2 or silicon nitride is rather inert to the oxidation process. After oxidation, a simple wet etch can remove the dielectric layer before the sample is loaded into the MBE chamber for epitaxial growth. We have performed such experiments and found that the threading dislocations actually increase during the lateral oxidation process. Residual film stress either tensile or compressive in the as-deposited dielectric layer is attributed to the increase of threading dislocations. So what we need for a cap protection layer is something less rigid than oxide or nitride and yet it can resist against wet oxidation. In addition, the cap layer needs to be amorphous or fine-grained polycrystalline so there is less constraint to the lattice match problem. Amorphous InGaP is one of such candidates. We have found that a relatively thin amorphous InGaP can be used for the surface protection during wet oxidation and it still allows for reduction of threading dislocations although the effectiveness of defect reduction decreases with an increasing thickness of the protection cap layer. The results are to be published.

Personnel supported

University of Illinois at Champaign-Urbana: 3 professors and 6 graduate students

The principal investigators contributing to various parts of the work reported here are professors (i) **K.C. Hsieh (PI)** (ii) K. Y. Cheng (iii) I. Adesida.

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(ii) Greg Pickell, Ph.D. student (Cheng advisor). (Ph.D. qualifying examination completed, 1998)

(iii) Hung Cheng Lin, Ph.D. student (Cheng advisor). (Ph.D. qualifying examination completed, 1999)

(iv) Kuo Lih Chang, Ph.D. student (Hsieh advisor). (Ph.D. qualifying examination completed, 1999)

(v) John H. Epple, Ph. D. student (Hsieh advisor). (Admitted to the graduate program in Fall, 1998)

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Sarnoff Corporation: 4 research engineers

The research engineers involved at the Sarnoff Corporation are

(i) John Connelly (ii) Ramon Martinelli (iii) Hao Lee and (iv) Michael Maiorov

IV. Publications/Conference Interactions

Publications: 7 published

(1) K.L. Chang, L.J. Chou, K.C. Hsieh, D.E. Wohlert, G.W. Pickrell, and K.Y. Cheng, "Formation of Amorphous Native-Oxides by Very-low-temperature Molecular Beam Epitaxy and Water Vapor Oxidation", J. Crystal Growth, 201/202, 633-637 (1999).

(2) D.E. Wohlert, H.C. Lin, K.L. Chang, G.W. Pickrell, Jr., J. H. Epple, K.C. Hsieh, and K.Y. Cheng,"Fabrication of a Substrate-Independent Aluminum Oxide-GaAs Distributed Bragg Reflector", Appl. Phys. Lett. 75, 1371 (1999).

(3) G.W. Pickrell, J.H.Epple, K.L. Chang, K.C. Hsieh and K.Y. Cheng, "Improvement of Wet-Oxidized AlxGa1-xAs (x~1) through the Use of AlAs/GaAs Digital Alloys," Appl. Phys. Lett. 76, 2544-2546 (2000).

(4) G. W. Pickrell, K. L. Chang, J. H. Epple, K. Y. Cheng and K. C. Hsieh,, "The Growth of Low Defect Density In0.25Ga0.75As on GaAs by Molecular Beam Epitaxy", J. Vacuum Sci. and Technol., B18, 2611-2614 (2000).

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Interactions/Transitions: 5 conference presentations

In addition to publishing in journals, we have also presented some of the research results in conference. Once of them is an invited talk.

1. K.C. Hsieh, "Aluminum and Gallium-bearing Native Oxide Formed by Wet-Oxidation of Amorphous Aluminum or Gallium Arsenide on III-V Compound Semiconductors", Materials Research Society, April 5-9, San Francisco, CA (1999), Invited Talk.
2. K.L. Chang, D.E. Wohlert, G.W. Pickrell, J. Epple, H.C. Lin, K.Y. Cheng, and K.C. Hsieh, " As Overpressure Mediated Crystallinity Change of AlGaAs Compounds and its Application in Formation of Bragg Reflectors", Electronic Materials Conference, June 30 – July 02, Santa Barbara, CA (1999).
3. G.W. Pickrell, K.L. Chang, J.H. Epple, K.Y. Cheng and K.C. Hsieh, "The Growth of High Quality In0.25Ga0.75As Epilayers on GaAs Substrates," Materials Research Society Symposium Proceeding, April, San Francisco, CA (2000).
4. K.L. Chang, J.H. Epple, G.W. Pickrell, , H.C. Lin, K.Y. Cheng, and K.C. Hsieh, " Strain Relaxation and Defect Reduction in InGaAs by Lateral Oxidation of AlGaAs Channel," 42nd Electronic Materials Conference, June, Denver, CO (2000).
5. J.H. Epple, K.L. Chang,, G.W. Pickrell, K.Y. Cheng, and K.C. Hsieh, "Thermal Wet Oxidation of Al0.4Ga0.6P," 42nd Electronic Materials Conference, June, Denver, CO (2000).

V. New discoveries, inventions and patent disclosures

None

VII. Honors / Awards

None